Coal Recycling from Tailings using Flotation with 2-Level Experimental Design Techniques

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Abstract
In this study, the possibility of producing coal with less than 11% ash from tailings of flotation process was investigated. The effect of six flotation parameters: collector type, collector consumption, frother type, frother consumption, pulp density and mixing rate were studied on a sample from a tailing dam. A software based experimental design approach (DX7) was applied to determine and model effective parameters as well as flotation optimization through fractional factorial. It was shown that collector type and flotation machine mixing rate were the most effective parameters on ash content of concentrate. The results indicated that the production of a desired ash content concentrate, i.e. <11% was feasible. It was also shown that at the optimum conditions of experiment, production of a concentrate with about 10% ash content, and 12% weight recovery would be possible.

Keywords: Coal Recycling, Tailings Flotation, Statistical Design

Introduction

Significant amounts of energy which are currently wasting in the form of tailings can be recovered by different mineral processing methods. Although coal tailings are used in civil engineering, agriculture and fluidized combustion most must be disposed in dumps off and slurry ponds, causing major economic and environmental problems [1-4] Among coal washing plant tailings, those from coal washing flotation circuit represent a significant part and may have considerable coal content, depending on the method used for coal washing as well as the size and distribution of fine mineral content in the coal. Fine coal tailings could have a calorific value in the range 7534.4-8790.2 kJ/kg [4].

Due to their high coal content, smaller size which increases the surface area liable to be wetted and oxidized, the dump of fine coal wastes in dams aggravates the risk of spontaneous combustion leading in turn to the emission of harmful gases. These adverse effects could be overcome by recovering coal from coal fines wastes prior to their disposal. Furthermore, the coal recovery from tailings reduces the need of coal to be extracted from mine and cost of handling and storage of coal wastes [4].

Extensive studies have been implemented and reported on coal recovery from washing plants tailings. For instance, column flotation has been implemented to recover coal from the flotation tailings and produce thermal coal [5-8]. Oil agglomeration is one of the applied methods for coal recovery from fine tailings too. For ash reduction of fine coals whose content has been increased in new extraction ways, agglomeration methods have also been studied [9-12]. To produce the coal which is used as coke in steel industries, one needs to use more precise mineral processing methods such

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as flotation practice by appropriate controlled conditions. Coal with less than 12% ash content and coke number of more than 7 is usually appropriate for conversion to suitable coke used in steel industries.

In this research work, the possibility of coal recovery from tailings of flotation circuits for producing coal with ash content of less than 12% by means of flotation method was studied. In order to understand and optimize the flotation process, a software based statistical methods (DX7) of experiments at two levels of parameters were applied.

Benefits of Applying Statistical Methods in Design of Experiments

Statistics provide a way to extract information from data [13]. For such purpose, statistical design of experiments is widely reported effective in the process characterization, optimization and modelling [14-16]. There are two approaches: i) one can investigate the effect of a large number of variables, and ii) the most commonly used method involves the variation of one variable while keeping the other variables constant (one factor at a time) until all variables to be studied. This methodology has two disadvantages: first, a large number of experiments are required, and second it is likely that the combined effect of two or more variables may not be identified [17]. The advantage of factorial design becomes more pronounced when more factors are used. The factorial design requires only 8 runs versus 16 for an OFAT experiment with equivalent power. Therefore using the 2-level factorial design, not only the number of runs is reduced, but also more information and well recognized is produced [13]

The number of experiments required for understanding all the effects is given by a^k where a is the number of levels and k is the number of factors [18, 19]. The full factorial approach for experimentation covers all combinations of factors, providing valuable information on interactions. However, the number of experimental runs increases rapidly. Fortunately, by resorting "fractional factorial" many factors could be studied and still keeping the experiments at reasonable size [13]. The designs are particularly useful when there are many factors and when the cost of experimentation is expensive [20]. Moreover, it is stated that only 20% of the main effects and two-factor interactions are likely to be significant in any particular system. If this is true, then only three effects will be significant, which leaves 28 effects for estimation of error, far more than necessary. Therefore, a full factorial on five factors or more will waste much of its unneeded estimate of error [13]. Consequently, the aim of the fractional (2^{k-m}) factorial design is to extract the part of experiments from the full factorial design which enables to obtain the main effects and some first order interaction [21]. Considering the above mentioned subjects, the implementation of flotation experiments with respect to several involved factors can be performed through fractional factorial to identify effective factors.

Material and Methods

Sample Preparation

A sample from the tailings of Central Alborz (Anjir-Tangeh) coal washing plant, which is located in north of Iran, were used for flotation tests. This sample was systematically taken, to provide for being the representative sample. Analysis of head sample showed an ash content of 48% on average and shale particles and some argillite, sandstone and clay as the main incombustible part. The results are shown in Table 1.

Normally ash part consists of Al_2O_3, SiO_2, TiO_2, Fe_2O_3, CaO, MgO, Na_2O and K_2O and other compounds that usually are associated with coal [22]. For the mentioned sample, ash content of +150 micrometers and -150 micrometers were

36.8% and 58.5%, respectively. The coke number depends on ash content, L.O.I and sulphur percentage of coal [23]. Plasticity properties play an important role in coal ranking and burning ability. For +150 and -150 micrometers, coke numbers of prepared sample were 4 and 1, respectively. Coal with coke number more than 5 is suitable for coke making process.

Flotation Tests

Two types of samples, ground and non-ground, with size distribution presented in Fig. 1, were used for flotation tests. The best coal recovery was obtained at pH=7 and slightly less [22]. Therefore, experiments at natural pH of sample were performed. According to Guadin, the ash content has an effect on coal hydrophobic property, because more ash means less hydrophobicity [24].

Table1: Coal characteristics parameters of sample

Fraction micrometer	Weight percentage (%)	Ash content (%)	L.O.I (%)	Coking Number	Plasticity properties	
					X (mm)	Y(mm)
+150	56.04	38.8	36.9	4	48	11
-150	43.96	58.5	44.8	1	-	-

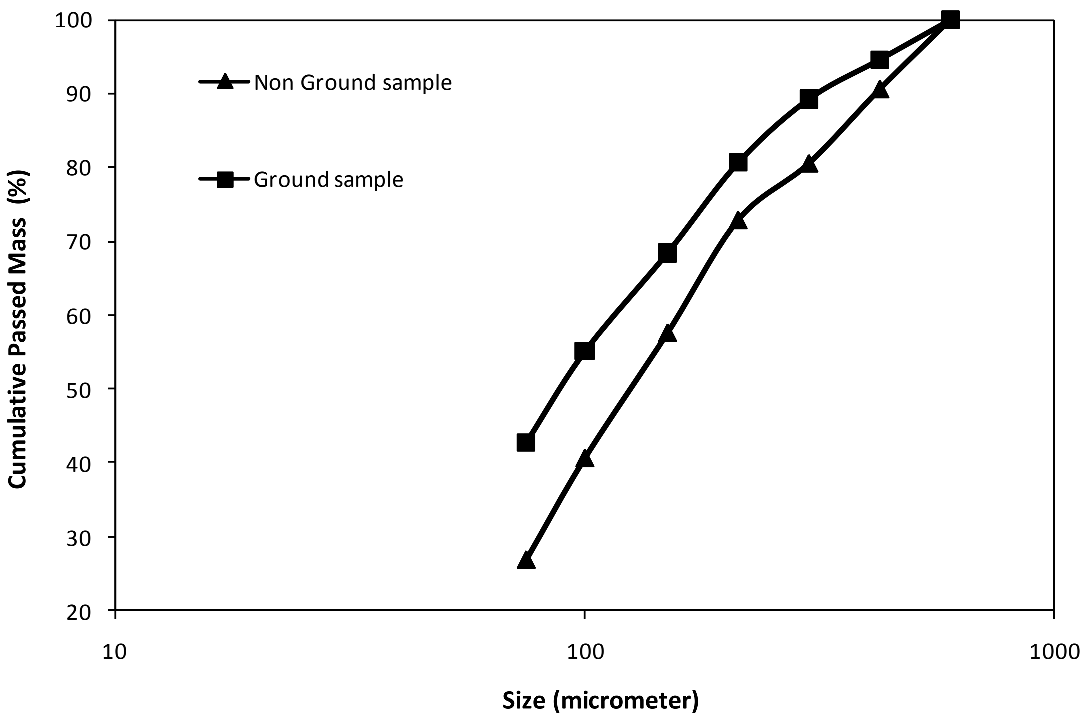

Figure 1: Size distribution of ground and non ground samples, used in flotation process

Table 2: Determined parameters limits for ground and non-ground sample (Mg means megagram)

Sample Type	Collector Type		Collector Amount (g/Mg)		Frother Type		Frother Amount (g/Mg)		Solid Percent (%)		R.P.M	
	A		B		C		D		E		F	
	Up	Low	Up	Low	Up	Low	Up	Low	Up	Low	Up	Low
Non-Ground Sample	Gasoline	Kerosene	800	200	MIBC	Pine Oil	180	110	20	14	1400	1100
Ground Sample	Gasoline	Kerosene	1000	200	MIBC	Pine Oil	200	110	20	12	1400	1100

Table 3: Designed experiments results for ground and non-ground samples of coal tailings

	Test status						Non Ground Sample			Ground Sample		
Test Number	A	B (g/ton)	C	D (g/ton)	E (%)	F	Conc. Ash (%)	S.E (%)	Com. Rec. (%)	Conc. Ash (%)	S.E (%)	Com Rec. (%)
1	A-	B-	C-	D-	E+	F+	32.23	47.60	85.51	34.22	43.44	49.28
2	A-	B+	C-	D-	E-	F-	25.02	50.87	66.75	24.01	53.01	23.48
3	A+	B+	C-	D-	E+	F-	20.44	26.56	28.93	29.22	51.69	33.84
4	A+	B+	C+	D+	E-	F-	22.26	45.93	55.12	27.65	52.25	30.15
5	A+	B-	C-	D-	E-	F+	28.03	48.87	69.18	21.23	29.56	9.50
6	A-	B-	C-	D+	E+	F-	29.18	50.76	77.31	28.81	50.97	32.85
7	A+	B+	C+	D-	E-	F+	23.25	49.97	62.64	32.08	47.95	44.85
8	A+	B-	C+	D-	E+	F-	25.50	41.81	52.12	27.65	50.38	30.21
9	A-	B+	C+	D+	E+	F-	31.42	48.73	81.46	32.08	48.67	41.83
10	A-	B-	C+	D-	E-	F-	27.07	51.02	71.49	26.65	54.65	31.35
11	A-	B+	C+	D-	E+	F+	32.54	44.63	88.83	38.02	36.68	55.26
12	A+	B+	C-	D+	E+	F+	31.27	48.64	80.56	26.78	47.64	25.18
13	A+	B-	C-	D+	E-	F-	27.58	43.09	55.85	27.14	52.24	30.03
14	A-	B-	C+	D+	E-	F+	32.13	47.79	81.34	32.47	47.89	44.44
15	A-	B+	C-	D+	E-	F+	35.96	40.34	89.04	38.88	35.68	59.37
16	A+	B-	C+	D+	E+	F+	29.30	51.94	81.04	31.85	47.19	43.29

Therefore, for plant tailing samples, the amount of collector consumption will be more than that of other coals. Calculating the optimum amount of reagents requires test implementation and statistical studies of their design. The effects of six parameters were studied in flotation process. If the tests were designed as full factorial, 64 (=2^6) tests would be performed for each sample. But as it is mentioned, the tests were designed and performed by fractional factorial approach with 16 (=2^{6-2}) tests. The lower and upper limits of six parameters were determined in preliminary tests. The results are shown in Table 2.

$$S.E. = \frac{c(f-t)(c-f)(100-t)}{f(c-t)^2(100-f)} \times 100 \qquad (1)$$

The relevant flotation tests were carried out in a Denver laboratory flotation machine model Q12. For each test 500g sample was used. After preparation and adding reagents, the concentrate was collected for 5 minutes and weighed to assess its ash content. Two parameters, that is ash content in concentrate and separation efficiency of combustible portion, were regarded as appropriate responses. The recovery was not used as a response, because it was not a clear factor of response modelling. Since recovery was never 100%, therefore separation efficiency (S.E.) was then used. Separation efficiency was calculated using relationship (1) in which f, c and t are the grades of coal in feed, and concentrate and tailing of flotation test, respectively. The results of experiments are given in Table 3.

4. Results and Discussion

Since the main objective of this research work was to study the possibility of recovering more than 50% combustible component from the tailing, thus S.E. was used, which is an index showing both the combustible and ash contents in the concentrate and also recovery of combustible part simultaneously. On the other hand to verify the creditability the experiments results, particularly to predict the other process conditions, a software based design (DX7) was used to develop the model of washing process in which the effective parameters are shown in a mathematical model. It is noteworthy that the factorial design was based on the notion that if all estimated effects were noise, they would have a normal (Gaussian) distribution and when plotted on a normal cumulative plot, would fall on a straight line. Hence, the effects significantly different from zero (noise) will fall outside the normal line [21]. Therefore, software was used to produce the probability plots and effective parameters regarding the considered responses, which are given in Figs 2 and 3. Having the effective response parameters determined, the appropriate models for creating each of these responses were established (Table 4). A brief ANOVA table for considered responses is also given in Table 5.

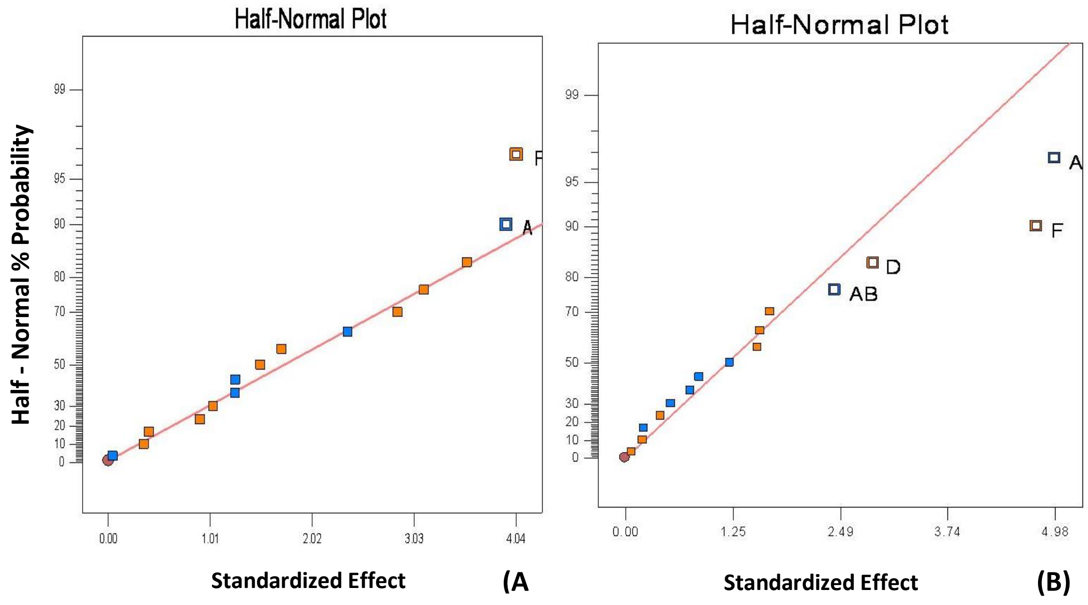

Figure 2: Standard effects on log-normal curve related to response of ash percentage of concentrate. A: ground sample, B: non-ground sample

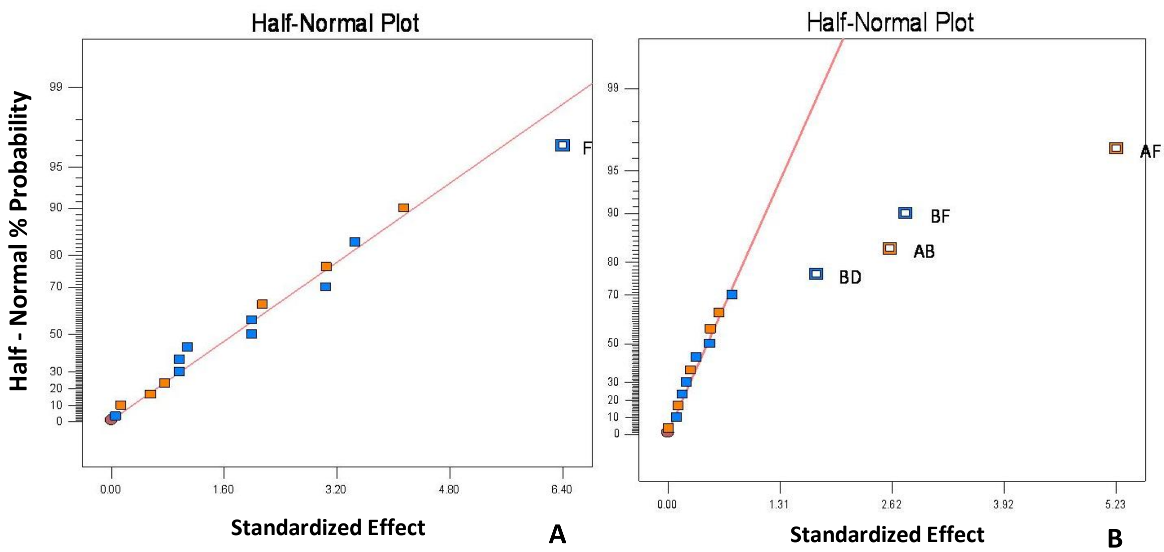

Figure 3: Standard effects on log-normal curve related to separation efficiency response. A: ground sample, B: non-ground sample

Table 4: The developed mathematical models for considered responses of ground and non ground samples (parameters are in the code form)

Considered Response	Non Ground Sample	Ground Sample
Concentrate Ash Content (%)	28.447-2.492A+1.440D+2.388F+1.22AB	29.920-1.970A+2.020F
Separation Efficiency (%)	47.398+1.297AB+2.615AF-0.866BD-1.386BF	48.531-3.201F

Table 5: ANOVA results of the equations for the studied responses by Design-Expert 7.0.0

Sample	Model	SD	CV%	R Squared	F Value	P Value (prob>F)
Non Ground	Ash model	2.00	7.04	0.85	15.44	0.0002
	S.E model	0.79	1.66	0.96	72.57	< 0.0001
Ground	Ash model	3.99	13.34	0.76	12.12	0.0063
	S.E model	4.8	10.06	0.75	13.90	0.0023

ANOVA makes it possible to check that the postulated model fits well the experimental points [21]. The model terms retained in the equations, are after elimination of insignificant variables and their interactions. A p-value of 0.05 or less rejects the null hypothesis "at the 5% level" that is only 5% of the time the supposed statistical model will fail to predict the response [25].

As to the p-values, it is accordingly stated that the probability of errors for proposed model for percentage of concentrate ash content and separation efficiency in non-ground sample were less than 0.02 and 0.01%, respectively. Similarly the probability of errors was less than 0.63 and 0.23% respectively for ground sample. For flotation tests, among the main factors for non-ground sample, parameters like collector type (A), the rpm of flotation cell (F), frother consumption, and collector type and consumption, interference on the percentage of concentrate ash content were identified to be effective. In ground sample, collector type (A) and rpm of flotation cell (F) factors were effective. For the both non-ground and ground samples, the rpm of flotation cells were identified as important factors. For concentrate of non ground sample, collector type and the rpm of flotation machine were the second factor with the highest effect. After these two parameters, frother consumption (D) interaction had more effects.

There is two ways to evaluate the model accuracy and performed implemented statistical method: i) residual normal plot, and ii) drawing the residual amounts against predicted values [13]. If the model could estimate the performed tests values with appropriate precision, the obtained residual values should have normal distribution. To do that, -1 should be considered for the low levels and +1 for the high levels in the model which is presented in following example. The model values for concentrate ash content and separation efficiency responses for the non-ground sample was calculated as below.

Concentrate ash content (%) = 28.447 - 2.492(-1) +1.440(-1) + 2.388(+1) + 2.02 (-1) × (-1) = 30.66

S.E.(%) = 47.398 + 1.297(-1)×(-1) × 2.615×(-1) × (+1) – 0.866(-1)×(-1) - 1.386(-1)×(+1) = 46.60

The obtained values of these two responses from the experiments were 32.23% and 47.60%, respectively, which had a deviation equal to 1.57 and 1.00 units from predicted values. Figs 4 and 5 show the absolute values of residual differences between test results and obtained response from the model. As it is seen, the obtained model for non-ground sample matches well with the performed experiments results. No particular trend in residual values against the test numbers indicates any systematic errors in performed tests. Systematic errors cause the residual values increase or decrease. Among all effective parameters, using both samples, the rpm parameter of flotation machine had the major effect on concentrate ash content response. Producing air bubbles and creating necessary conditions for attaching coal particles to air bubbles, and reaching to the surface of pulp and collecting them, were the necessities for successful flotation. In laboratory flotation machines, this factor determines the rate of mixing and cell turbulent, and has an effect on the inlet air flow [22]. The collector type parameter was chosen because of the differences between kerosene and gasoline. Kerosene represents more selective behavior rather than gasoline, but gasoline is more hydrophobic than kerosene [22].

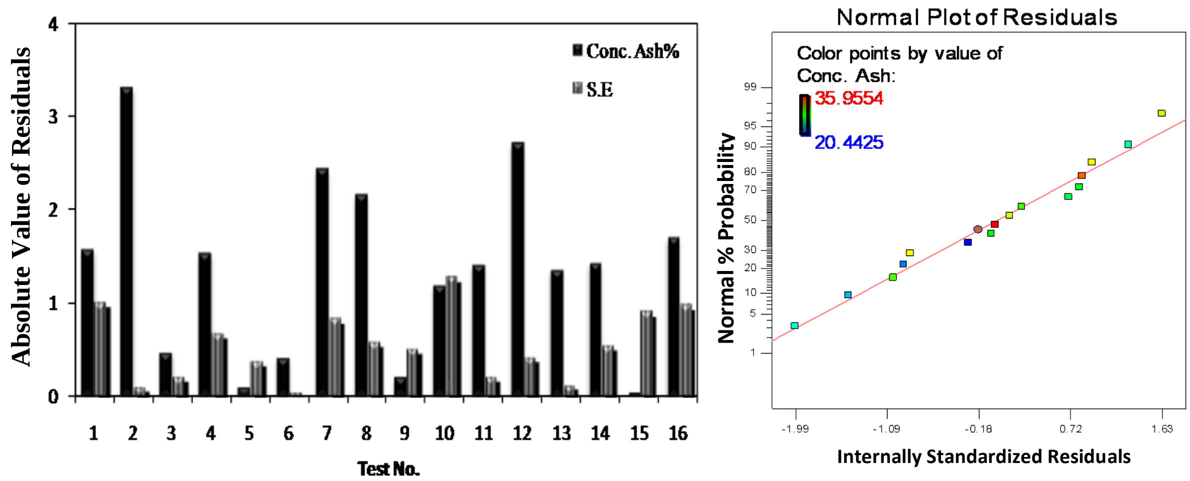

Figure 4: Normal graphs of residual values (right), residual values of responses from performed tests and obtained results from model for non ground sample in 16 performed tests (left)

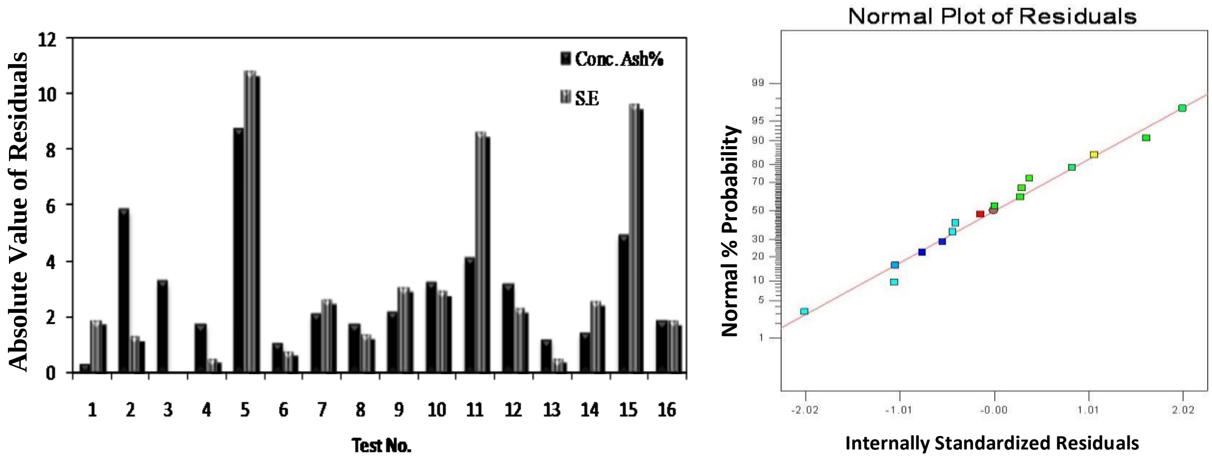

Figure 5: Normal graphs of residual values (right) residual values of responses from performed tests and obtained results from model for ground sample in 16 performed tests (left)

Table 6: The optimum conditions determined by software for performed tests, along with predicted responses (minimum ash content of concentrate and maximum separation efficiency for ground and not ground samples)

Sample Type	Collector Type	Collector Consumption (g/Mg)	Frother Type	Frother Consumption (g/Mg)	Solid Percent (%)	rpm	Ash Content of Concentrate (%)	Separation Efficiency (%)	Degree of response desirability
Non Ground Sample	Gasoline	800	MIBC	110	16	1100	22.12	53.33	92.67
Ground Sample	Gasoline	1000	MIBC	170	14	1100	25.93	51.71	84.60

Table 7: Optimization criteria for different variables and responses

Variables/Responses	Goal	Importance
Collector (Kerosene/Gasoil)	In the range	1
Collector Value (g/Mg)	In the range	1
Frother (Pine Oil/MIBC)	In the range	1
Frother Value (g/Mg)	In the range	1
Solid Percent (%)	In the range	1
Rpm	In the range	1
Ash content of concentrate (%)	Minimize	3
S.E. (%)	Maximize	3

Process Optimization

Numerical optimization technique was used for simultaneous optimization of multiple responses [25]. After modeling the responses, then conditions of optimal experiment were accordingly obtained. The considered conditions for responses and parameters are given in Table 6. The values of "Importance" in Table 7, denote the weight of each variable and response, and the effect of each variable on the responses (which were observed after performing ANOVA analysis). The effect of level changes of effective factors on the response of concentrate ash content is given in Fig. 6.

The effect of gasoline usage as collector against kerosene in both fractions was determined. Also with rpm increase, the obtained concentrate ash increases and for non ground sample, less frother should be used. Applying the considered conditions and using DX7 software, optimal conditions were obtained for both ground and non-ground samples. In order to explore a solution maximizing multiple responses, the goals were combined into an overall composite function, called the desirability function [26]. Desirability is an objective function that ranges from "0" for least desirable, to "1" for most desirable. The numerical optimization finds a point that maximizes the desirability function [25].

The process interpretation will be possible more easily by observing the optimal conditions which were proposed by the software. In general, gasoline was a better choice rather than kerosene in order to make the coal particles more floating with more hydrophobicity properties. Turbulence and disturbance in environment should be set to the lowest rate (Figs 6-c, 6-e). For this sample, gasoline reduced the concentrate ash, and its consumption should also be adjusted to the highest levels (Fig 6-a). With respect to different applied rpm's, bubble levels were independent from speed and how they enter to the pulp. But it was remarkable that the ascent rate of air bubbles increases as the machine's rpm increases [23]. However, it happened as a response to an increase of fine particles which constitute the main non-combustible part in this section, the increase of air bubbles movement in pulp, caused an increase in ash content of concentrate. So, the flotation machine rpm was as the lowest level (Figs 6-d, 6-f). According to Fig. 6, the collector type and rpm have maximum effects on response of concentrate ash for both samples. The lowest concentrate ash was obtained, when gasoline used as collector and rpm was equal to 1100. For non ground sample, frother amount was also at its lowest level (Fig. 6-b).

The dose of 800 g/Mg gasoline as collector and 110 g/Mg MIBC as frother was suggested by the software for non ground sample. Also the solid percent and flotation machine rpm were proposed to be 16 and 1100, respectively. The software predicted that applying these conditions, a product with ash content of 22.12% and 53.33% of S.E. would be obtained. The proposed conditions by the software, for ground sample using 1000 g/Mg gasoline and 170 g/Mg MIBC, were: the solid percent and flotation machine rpm equal to 14% and 1100 respectively. The software predicted that by applying these conditions, a product with ash content of 25.93% and the separation efficiency of 51.73% will be obtained.

Model Validation

Flotation experiments under the calculated conditions were performed, using both samples to evaluate the validation of the models. Their results are presented in Table 6. For each sample, the final optimal tests was then repeated 4 times and the obtained products were then mixed and washed at two stages in order to investigate the possibility of achieving a concentrate with ash content of less than 12%. The tests results are given in Table 8. For the non ground sample, the average amounts of concentrate ash and separation efficiency were 26.43% and 55.17%, respectively. If the mean obtained values resulting from the optimizing experiment, the concentrate ash is more than the predicted value by 4.31 (26.43-22.12) units, and obtained separation efficiency is different from predicted value by 3.46 (55.17-51.71) units. For the ground sample, the difference between obtained concentrate ash and predicted one is 2.33 (28.26-25.93) and the difference for separation efficiencies is 0.13 (51.84-51.71). After washing stages of mixed concentrates, the results indicated that it is not possible to reach 12% ash content of concentrate from the non ground sample (after two washing stages). Unlikely, the ground tailing sample after second washing stage will reach the desired concentrate quality with ash content of less than 12%.

The results from this investigation that using the non ground sample to achieve a concentrate with less than 12% ash was not suitable, and it needs more grinding to create new surfaces and size fractioning. Coal recovery values indicated that the final product (second cleaner concentrate) for ground sample had more ash in comparison to the non ground sample. Recovery of combustible part of the non ground sample in first flotation stage (rougher) was 81.58%, while it was 82.5% for the ground sample. The difference was clear in washing stages so that it was 33.15% for the non ground sample (in the first stage) and 47.3% for ground sample. It was 15.26% for the non ground sample (in the second stage), while it was 21% for the ground sample. Thus, it can be stated that the sample should be ground for producing suitable coal for coke industries with ash content less than 12%. Flotation test (after two-stage washing) could produce a concentrate with weight percent and ash content of 12.32% and 10.11%, respectively

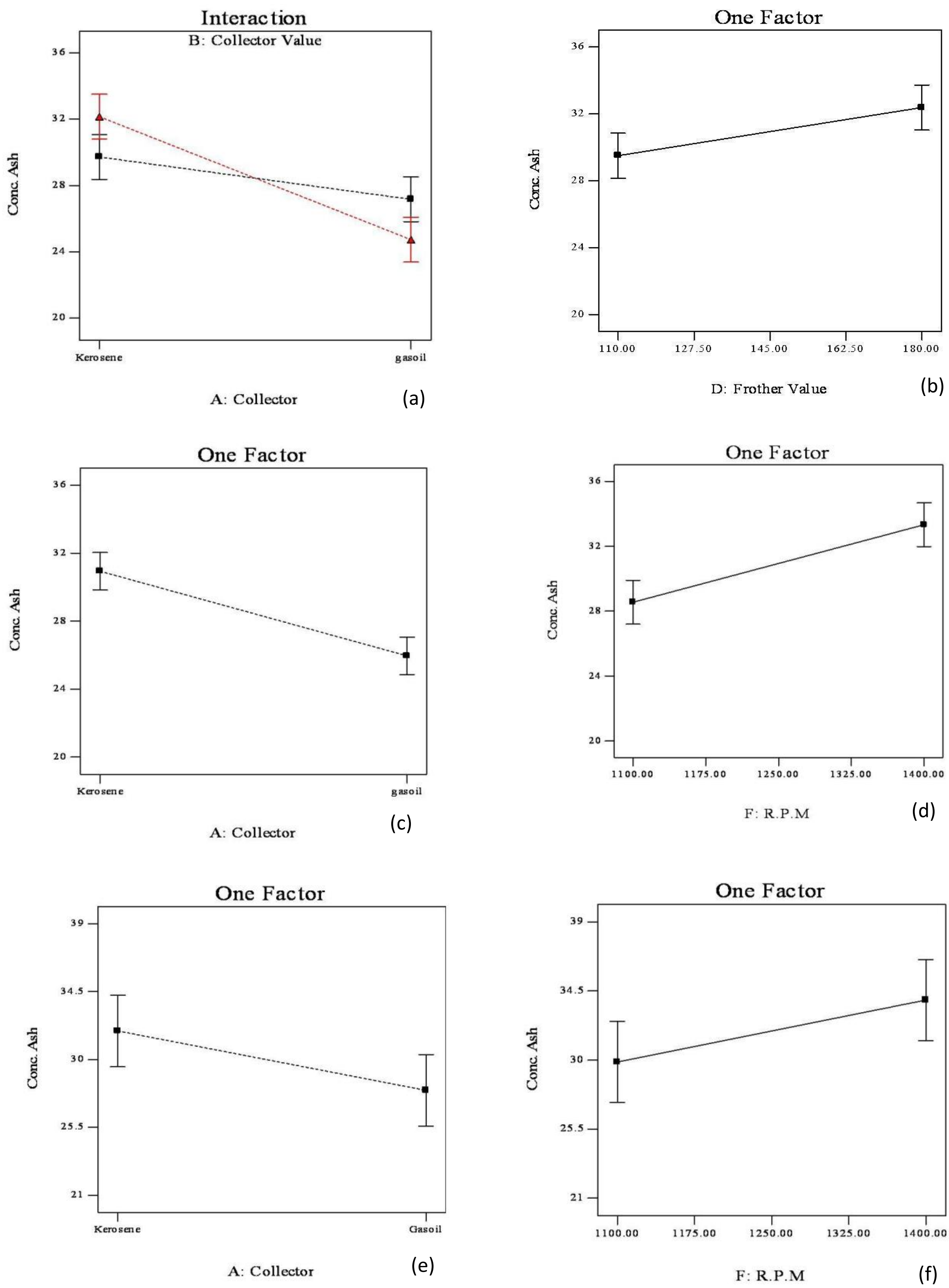

Figure 6: Effect of effective parameters on concentrate ash: A) non ground sample, B) ground sample. Choosing gasoline for both samples causes a decrease in ash content. The increase of rpm increases the product ash and less amount of frother should be used for non ground sample.

Table 8: Proposed model validation using independent experimental results

Sample	Rougher stage					First Cleaner Stage					Second Cleaner Stage			
	wt (٪)	Ash Content of Conc. (٪)	S.E (٪)	Coal Rec. (%)		wt (٪)	Ash Content of Conc. (٪)	S.E (٪)	Coal Rec. (%)		wt (٪)	Ash Content of Conc. (٪)	S.E (٪)	Coal Rec. (%)
Non Ground Sample	58.95	23.67	52.03	81.58		21.7	18.61	30.17	33.15		9.5	14.60	21.73	15.26
		26.66	57.38											
		27.48	56.26											
		27.89	55.01											
	Aver.	26.43	55.17											
	S.d	1.91	2.31											
Ground Sample	61.13	26.89	50.88	82.5		29.5	15.32	38.88	47.3		12.3	10.11	31.97	21.0
		27.9	52.51											
		28.72	49.02											
		29.54	54.93											
	Aver.	28.26	51.84											
	S.d	1.13	2.50											

Conclusions

In this research the possibility of producing a concentrate with less than 12% of ash from coal flotation tailings was studied. The average ash content of the used samples for flotation experiments was about 48%. Tests were performed using two non ground and ground samples, and fractional factorial approach. For the non ground sample, statistical studies results indicated that three factors such as collector type, rpm of flotation machine speed, frother consumption and interaction of collector type and its consumption had significant effects on the responses. Two factors such as collector type and rpm of flotation cell had effects on concentrate ash content, using ground sample. It could accordingly be stated that:

- gasoline diesel oil was better for covering the destructed surfaces of coal particles and flotation than oil (kerosene)
- stirring intensity should be set to the lowest. An increase in the environment turbulence causes coal particles to be separated from air bubbles. In laboratory scale, increasing rpm flotation cell increased the ascent rate of air bubbles, and more fine particles reported to concentrate and accordingly increased the ash in product.

The validation test was performed by applying optimal conditions for the non ground sample, including 800 g/Mg gasoline as collector, and 110 g/Mg MIBC as frother as well as 16% solid percent and 1100 rpm. A concentrate was obtained with ash content and separation efficiency of 26.43% and 55.17% respectively. Similarly, proposed conditions by software for the ground samples was 1000 g/Mg gasoline, 170 g/Mg MIBC, 14% solid percent and rpm equal to 1100, which, applying this condition, provided a product with ash content and separation efficiency of 28.26% and 51.84%, respectively.

Two washing stages were performed on the obtained concentrates from the non ground and ground samples and obtained products had ash contents of 14.60% and 10.11% respectively. Comparing flotation products of the non ground and ground samples under optimum conditions indicates that sample deposition in tailing dams, leads to destruction in coal particle surfaces due to oxidation and it is hard to produce concentrate with less than 12% ash content by flotation, while with only one grinding stage and creating new surfaces (by grinding), after two concentrate washing stages, the obtained concentrate would have the desired ash content.

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